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Coláiste na hOllscoile Corcaigh

Dilute Phase Pneumatic Conveying of Whey Protein Isolate Powders: Particle Breakage and its effects on bulk properties

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Abstract: Breakage of dairy powder during pneumatic conveying negatively affects the endcustomer properties (scoop uniformity and reconstitution). A dilute phase pneumatic conveying system was built to conduct studies into this problem using whey protein isolate powder (WPI) as the test material. Effects of conveying air velocity (V), solid loading rate (S_L), pipe bend radius (D), and initial particle size (d) on the level of attrition were experimentally studied. Four quality characteristics were measured before and after conveying: particle size distribution, tapped bulk density, flowability, and wettability. The damaged WPI agglomerates after conveying give rise to many porous holes exposed to the interstitial air. V is the most important input variable and breakage levels rise rapidly at higher airspeeds. The mean volume diameter D[4,3] decreased by around 20% using the largest airspeed of 30 m/s. Powder breakage is also very sensitive to particle size. There appears to be a threshold size below which breakage is almost negligible. By contrast, S_L and D show lesser influence on powder breakage. Reflecting the changes in particle size due to breakage, tapped bulk density increases whereas wettability decreases as a result of an increase in conveying air velocity. However, breakage does not show a significant effect on powder flowability as powder damage not only decreases particle size but also changes the particle's surface morphology.

Keywords: pneumatic conveying; powder breakage; wettability; flowability; whey protein isolate. **PACS number:** 45.70.MG – granular flow

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1. Introduction

In dairy processing, pneumatic conveying is typically selected for transporting dairy powders either from a fluidized bed or directly from the spray dryer to the storage silo or bag filling stages of the manufacturing process. Industrial pneumatic conveying systems can be classified as being either lean phase or dense phase and each, in turn, can be operated with positive pressure ('pushing') or vacuum ('pulling'). In the dairy sector, all systems use stainless steel piping for sanitation and corrosion resistance. Powder particles undergo multiple impacts on the pipe wall (especially at the bends) during pneumatic transportation ^[1], which will cause breakage of powder after conveying. Attrition of dairy powders changes the size, shape, and structure of the powder.^[2] This negatively affects the handling of the product (wettability and flowability) and end-customer properties (scoop uniformity, reconstitution, etc.)^[3, 4]. Wettability is a measure of how readily a powder can absorb water. Poor wetting powders tend to float on the surface of still water and sink very slowly into the water, which will directly affect the rehydration of dairy powders which is a very important functional property of dairy powders.^[5] Powders that have undergone large amounts of breakage, resulting in a smaller particle size tend to be less easy to wet.

There are a number of studies which are devoted to (food) powder breakage in pneumatic conveying systems ^[6-8]. Many factors are found to play a role in powder attrition. Kalman ^[9] and Aked ^[10] confirmed the considerable influence of the number of bends on the breakage level. Kalman ^[11] also demonstrated the influence of vibration of bends on powder attrition. Konami et al. ^[12] focused the investigation on changes in the size of granules in the attrition process during repeated pneumatic transport. With the aim of identifying the basic breakage mechanisms, some experiments^[13, 14] based on well-defined conditions in simple set-ups were carried out. In addition to experimental methods, some simulation approaches combined Computational Fluid Dynamics (CFD) with Discrete Element Method (DEM)^[15-19] to explore

the degradation and dynamics of particles in conveying systems.

In contrast to the considerable effort that has been made to examine the pneumatic conveying of food or pharmaceutical powders, there are few works have been carried out in the specific area of transporting dairy powders. Hanley et al. ^[20,21] investigated the effects of conveying conditions and conveying mode (dense or dilute phase) on attrition of infant formula in a lab-scale modular pneumatic conveying rig with a diameter of 25 mm. They found that mode of conveying, conveying air velocity and a number of passes all had a statistically significant effect on bulk density. For mean volume diameter (D[4,3]) and wettability, the mode of conveying was the only significant factor, while none of the factors had a statistically significant influence on particle density. Boiarkina et al.^[22] focused research on the conveying of Instant whole milk powder (IWMP) at two industrial plants with different transport systems; a pneumatic system and bucket elevator. They evaluated the importance of breakdown of the final product properties given different conveying methods and suggested that producing powders of the right agglomerate size and bulk density prior to transport can compensate for the inevitable particle breakage.

The dairy industry conveys a huge range of powder through pneumatic lines. In general, fine dairy powders (average diameter smaller than 500 micron) have unique properties due to the complex components, especially the fat content, lactose, and protein content.^[23] This makes the conveying behavior of dairy powder considerably different from ordinary food powders which normally have a relatively much bigger size. Whey protein isolate (WPI) is the purest form of whey (protein content \geq 90%) and is a complete protein. It contains all the essential amino acids that the human body needs to repair muscle after a workout. In this work we conducted experimental research on pneumatic conveying of whey protein isolate (WPI) powders in a laboratory-scale dilute conveying system with positive pressure, and probe the effects of conveying operating conditions (conveying airspeed and solid feeding rate), bend

radii, and initial particle size on the breakage level of WPI. To quantify their influences on the change in powder properties, particle shape, and surface analysis is employed and some quality characteristics are measured before and after conveying: particle size distribution, tapped bulk density, wettability, and flowability. This is the first study to comprehensively examine the pneumatic conveying of dairy powder in an experimental unit that relates to industrial practice.

2. Materials and Methods

A lab-scale pneumatic conveying rig (dilute phase, positive pressure), with 50 mm diameter food-grade 316L stainless steel pipes (1.5 meters high and 5 meters conveying distance with three 90° bends, two in the vertical plane and one in the horizontal plane) was built as the test system, as shown in figure 1. A long horizontal pipe is connected with a vertical pipe using an upward bend, and another bend turns the direction of pipelines from vertical into horizontal again. The last bend connects two horizontal pipes. All the three bends have the same radius, 300 mm. An electric vibratory feeder (DR 100 Retsch, Germany) is used to load powder particles continuously with a changeable feeding rate. The system also contains a pitot tube for measuring supplied air velocity and a pressure meter (Digitron Pressure Meter 2022P, 2 bar operating range, 4 bar over range) for determining the pressure drop during conveying.

Whey protein isolate (90% Protein, WPI) powders provided by one typical Irish dairy company were used as the test material. The WPI samples are produced by spray drying followed by fluidized bed drying. Specifically, WPI powders with five different initial size ranges (0-1000 μ m, 25-250 μ m, 250-425 μ m, 425-850 μ m, 700-1250 μ m) were used. The corresponding mean volume diameter D[4,3] for these ranges is 338, 189, 352, 657, and 921 μ m, respectively. Dry compressed air was used for conveying and its pressure could be regulated to provide air velocities of up to 30 m/s. At the pickup point, the temperature of the air is 18 °C at a relative humidity of 25%, and the operating pressure is between 0.25 to 1.4 bar based on different air velocities used. For every trial, a 100 g batch of WPI powders at a given feeding rate was fed into the funnel and blown through the pipe at a given conveying velocity. Three values of conveying airspeed (10 m/s, 20 m/s, 30 m/s), and four solid feeding rates (2.5 g/s, 10 g/s, and 20 g/s) were used in this study. Correspondingly, the solid loading ratio (the mass flow ratio of solid to conveying air) was in the range from 0.08 to 1.02, which is consistent with the range of solid loading conditions used by some dairy companies. The minimum conveying air velocities at the pickup point (U_{pu}) for the WPI powders used are varied depending on the solid loading used, however, all the (U_{pu}) for all the solid loading conditions explored are lower than 10 m/s. The operation parameters (conveying air velocity range and solid loading ratio) investigated in our study are set by the need to avoid line blockages (lower limit) and test system capability (upper limit), and it also covers the operating conditions to mimic the actual transportation practice of the company.

To quantify the change in powder properties, four quality characteristics are measured before and after conveying: particle size distribution, surface analysis, bulk density, and flowability and wettability. The volume means diameter D[4,3] and median diameter D_{50} was used as the measures to compare particle size result.

2.1 Scanning electron microscope (SEM)

Some representative WPI samples were visualized using a scanning electron microscope (SEM). Samples for analysis were mounted on double-sided carbon tape, attached to SEM stubs, and then sputter-coated with chromium. The accelerating voltage used for the SEM was constant at 5 kV, while the magnification was varied from 200X to 3000X.

2.2 Measurement of Particle size distribution (PSD)

Particle size distribution of the powder samples before and after conveying was measured by a mechanical tapping sieve shaker with 10 particles size mesh (50 μ m to 1000 μ m). The powder samples were weighed using the weighing balance and poured into the top sieve. After being vibrated with the same strength and time, the different aggregate of particle sizes was collected on each sieve and weighed. The particle size distribution was measured twice for each trial to increase accuracy. Particle size distributions were calculated based on results from sieve analysis.

2.3 Measurement of tapped bulk density

Bulk density of powder samples was measured using a Stampfvolumeter STAV 2003 (J. Engelsmann AG, Luwigshafen, Germany). For each measurement, 100 ml of powder was poured into the graduated cylinder and tapped 1250 times to the extreme powder bulk density. Bulk density was measured twice for each trial to increase accuracy, and the mean result was used for analysis.

2.4 Measurement of wettability

Wettability is the ability of the powder particles to imbibe a liquid and overcome the surface tension between them based on capillary force. Generally, the GEA Niro method^[24] is used to measure the wettability of "easy to wet" powders. However, for some powders with poor wetting ability, such as WPI powder in this study, it can be determined by the additional weight of powder after absorbing liquid in a specified time, known as the Washburn method.^[25] In specific, we quantified the wettability of WPI powders by measuring the weight of adsorbed water in 2 g powders after 10 min.

2.5 Measurement of flowability

A Powder Flow Tester (PFT) from Brookfield (Brookfield Engineering Laboratories, Inc., Middleboro, MA, USA) was used for determining the flowability of WPI powder samples. Axial and torsional speeds for the PFT were 1.0 mm s⁻¹and 1 rev h⁻¹, respectively. WPI powders were filled into the aluminum trough of the annular shear cell (5-inch diameter) at room temperature (20°C). Flowability was measured using standard flow function test, involved the application of 5 uniaxial normal stresses (between 0.1 and 11 kPa) and 5 over consolidation stresses at each normal stress. The standard flow function test for each kind of powder sample was repeated 3 times.

3. Results: Powder breakage

3.1. Effect of conveying air speed

Figures 2(a) to (d) show micrographs of WPI powder samples taken at different magnifications. WPI powder before conveying (figure 2a) has a relatively bigger size and as well is an agglomerated powder with rough and complex surfaces. It also can be seen that there is no visible damage to the overall structure or of the component particles for it. On the contrary, Figure 2(b) and (c) show the clear damage and decrease in particle size after conveying, at a conveying velocity of 10 m/s (Figure 2b) and 30 m/s (Figure 2c), respectively. Also, the complete breakage of a single particle is shown in figure 2(d). In addition, the damaged particles contain many porous holes exposed to the interstitial air compared to the intact particles before conveying.

Figure 3(a) shows the particle size distributions of the powder in relative frequency form before conveying and after conveying using three different airspeeds. The solid loading rate was fixed at 5 g/s. It was found that the particle size distribution changed after conveying with a conveying airspeed (V) of 10 m/s. Powder breakage levels rise as the airspeed increases to 30 m/s. Specifically, as air speed increases, a greater proportion of the powder lies within the smaller size classes. Figure 3(b) plots mean volume diameter D[4,3] as a function of air speed; mean diameter falls significantly with increasing air velocity. For example, D[4,3] is reduced from 338 µm to 258 µm after conveying at the highest velocity of 30 m/s; the decrease is around 20%.

The important effect of air speed on the level of powder breakage can be further analysed

by examining the cumulative size distribution of the powder, before and after conveying, in graphical form. Three breakage parameters can be defined from the graph. The first is breakage potential, B_p , which is theoretical maximum amount of breakage that can occur (if all particles breakdown into infinitesimally sized fragments). Graphically it is represented by the cumulative shaded area on the left side of the dashed line showing the particle size distribution before conveying on as illustrated in figure 4a. The actual amount of breakage (B_t) that occurs is indicated by the shaded area between the cumulative distribution before conveying and after conveying (illustrated in figure 4b-d). Finally relative breakage, B_r is defined as the ratio of actual breakage to breakage potential i.e. B_t/B_p . The calculations of the integration area were made numerically for the different conveying speeds and the data given in table 1. Clearly as air speed increases, more of the potential breakage actually occurs.

3.2. Effect of solid feeding rate

Figure 5 shows the effect of solid loading rate on powder breakage at the fixed conveying airspeed of 30 m/s. Particle size distribution before conveying and after conveying at three loading rates (2.5, 5 and 20 g/s) is shown. It can be seen that increasing the solid loading rate can reduce the breakage of WPI to a certain degree, but the positive effect is not significant and small compared to the effect of air speed. Breakage of WPI powder is still considerable after conveying even with the largest feeding rate 20 g/s (corresponding to a solid loading ratio equals to 1.02).

3.3. Effect of bend radius

In any pipeline, the severity of the bend affects the level of powder breakage. To explore this, three different bend radii (R) were tested; 200 mm, 300 mm and 400 mm respectively. For each of the three tests, the line configuration was two 90° bends in the vertical plane separated by a straight section 900 mm long. The three configurations are shown in figure 6; figure 6a

shows the two 200 mm radius bend (bend radius to pipe diameter is 4), figure 6b shows the two 300 mm bends (bend radius to pipe diameter is 6) while figure 6c displays the two 400 mm bends (bend radius to pipe diameter is 8). The systems are shown in figure 6a and 6b correspond to short-radius bends, while figure 6c is using long radius bends.

Figure 7 shows the particle size distribution of WPI before conveying (dashed line) and WPI after conveying with the conveying condition where conveying air velocity is 20 m/s and solid feeding rate is 5 g/s. Unsurprisingly it was found that the greatest powder damage is achieved by using two bends with the smallest radius, 200 mm. The size distribution data also shows that for the gentler bends, a greater proportion of the powder remains in the larger size classes. This is because in addition to the conveying conditions (conveying speed and solid loading), particle impact angle (α) also is a very important variable with respect to the dilute conveying system and α will strongly depend on the radius of the bend (R). In a short radius bend, the particles will impact at a high (sharp) value of the angle. However, in a long radius bend, the impact angle (α) will be much lower (giving a more gentle impact) which is helpful in reducing attrition.

3.4 Effect of initial particle size (distribution)

Figure 8 shows the particle size distribution of WPI powders before and after conveying with using four different initial size ranges (25-250 μ m, 250-425 μ m, 425-850 μ m, 700-1250 μ m) and correspondingly the mean volume diameter D[4,3] of 189, 352, 657, and 921 μ m.

It can be seen from figure 8 that, under the same operating condition (conveying air velocity V = 20 m/s and solid feeding rate S = 5 g/s), WPI with a large initial size range (700-1250 µm) experienced significant attrition; however, there is a significant reduction in breakage rate with decreasing initial particle size. For the powder sample with the largest initial size, the mean volume diameter D[4,3] reduced from 921 to 296 µm. The breakage rate is up to 60%. Whereas,

there is almost no obvious damage of WPI with the smallest initial size (189 μ m). D[4,3] after conveying is 180 μ m, therefore the breakage rate is less than 5%.

The reduction in mean diameter with increasing air speed for the four different powder size classes is shown in figure 9. Compared to powder with smaller particle size, the change in the particle size distribution of powder after conveying with the large initial size is more sensitive to conveying air velocity used. There is a dramatic decrease of D[4,3] for the powder sample with a large size range 425-850 μ m and 700-1250 μ m with increasing the conveying air velocity. However, powder with a small initial size (25-250) appears not very sensitive to conveying air velocity. For this powder, there is only very limited change in D[4,3] with increasing conveying air velocity from 10 to 30 m/s.

In summary, for a given line configuration and conveying conditions, larger-sized particles break more readily than smaller sizes, and furthermore are more sensitive to changing conveying conditions. In addition, it can be concluded that there is a threshold value of initial particle size (range) smaller than which there is little powder breakage/attrition after pneumatic conveying.

4. Results: Powder Bulk Density, Wettability & Flowability

4.1 Tapped bulk density

Tapped bulk density is one of the most important dairy product properties dependent on powder size. Figure 10 shows the effects of operating conditions (airspeed and solids loading rate) on WPI powder tapped bulk density; figure 10a gives the values of bulk density and figure 10b the percentage change in value. Clearly, at a fixed solid feeding rate of 5 g/s, tapped bulk density of WPI powder increases with higher conveying air velocity as a consequence of greater breakage induced by stronger impacts. It also was found that in general bulk density increased with a lower solid feeding rate. However, the tapped bulk density of WPI is more

sensitive to changes in conveying airspeed rather than that of solid feeding rate. It also can be seen from figure 10b showing the percentage change in bulk density that there is a considerable increase in tapped bulk density (almost 4%) of WPI at a fixed solid feeding rate of 5 g/s while the conveying air velocity increases from 10 to 30 m/s (an increase of 3 times). However, the variation of solid feeding rate only has a relatively small influence on bulk density. Even with the highest velocity (30 m/s), the maximum change in bulk density is only around 2% with the variation of feeding rate from 2.5 to 20 g/s (an increase of 8 times).

4.2 Flowability

The shear cell test result (figure 11) shows that the flowability of WPI powders before and after conveying did not differ significantly. Before conveying, the powder was found to have a flow index of 5.8 while after conveying it lay between 5.0 and 5.6 depending on the magnitude of the conveying air speed. There was no consistent pattern between flow index and air speed. According to the flow index proposed by Jenike^[26], all the powder samples before conveying and after conveying can be classified as easy flowing; however, all three powders after conveying have a smaller flow index value which indicates that powders become more slightly cohesive after conveying. This can be attributed to the decrease in particle size resulting from breakage of agglomerates during conveying process. The decay in particle size could tend to reduce powder flow due to the increased surface area per unit mass has provided a greater surface area for surface cohesive forces to interact resulting in a more cohesive flow.^[27] Although after conveying the powder is a little more cohesive, the changes in flowability are not very significant. Powder flowability is influenced by powder composition, and other physicochemical properties, such as particle size and microstructures (morphology).^[2, 28] Powder breakage caused by pneumatic transportation does not change the powder composition. After conveying particle size decreases due to breakage but breakage also affects particle morphology because of both impact and surface polishing effects at the pipe bends. Highvelocity pneumatic conveying creates a large number of damaged particles which also have a more regular shape and smoother surface (seen in figure 2), compared to the aggregated unbroken particles. The combined effects of decreasing particle size and improving particle surface smoothness shaped the flow behavior of the WPI particles after conveying.

4.3 Wettability

The wettability comparison between WPI powders before conveying and transported by using different conveying conditions is present in figure 12, where the wettability is quantified by measuring the weight of adsorbed water. There is a decrease in the amount of adsorbed water into the powder as the conveying air velocity is increased from 10 to 30 m/s (see figure 12a) with the fixed solid feeding rate of 5 g/s, There is a slight increase in the amount of absorbed water as the solid feeding rate increases from 2.5 to 20 g/s (see figure 12 b) with a fixed airspeed of 30 m/s. Clearly, the pattern of wettability reflects the pattern of powder size. The impact that particles experiences with high conveying velocity transportation (or less solid loading) not only largely reduces the sizes of WPI powder particles but also created the fine particles with low porosity due to the formation of smaller void spaces between the fine damaged particles ^[29]. By the contrary, larger size WPI powders undergoing less or lower impact at lower conveying air velocity (or higher solid loading) have better wettability as the water is more easily able to permeate between the powder particles with the larger void space and wet them more quickly.

5. Conclusion

In this study, WPI powders were transported in a dilute phase, positive pressure conveying system. The effects of air velocity, solid loading rate, bend radius and initial particle size on powder attrition during transportation were explored. Powder integrity could be greatly improved by decreasing conveying velocity or selecting particles with a smaller initial size. Increasing solid loading and using longer bends also can make a contribution to reducing breakage; however, the effect is not as significant as the former two effects.

Breakage of WPI powders during pneumatic conveying will result in changes in properties of powders, particularly the particle size distribution, tapped bulk density, and wettability. Specifically, tapped bulk density increased while wettability decreased due to the greater breakage induced by high conveying air velocity. However, it seems flowability is not significantly affected by the breakage of powder particles. This may due to that powder breakage leads not only to decreased particle size but also to change the particle's surface morphology; the smoothing of the particle surface may partially compensate for the reduction in particle size. Overall the study has shown that optimizing conveying system/conditions is not the only way to control the effects of breakage, and the focus should equally be on producing powders of the right agglomerate size (smaller and narrow distribution) and bulk density prior to transport, such as in the spray dryer. Of course, powder particle size and its distribution is informed by and affect the whole manufacturing and distribution process, and the particle size distribution impacts on a large number of product quality parameters, so it cannot be selected solely with pneumatic transport in mind.

Finally, while this work has focused on one type of dairy powder, many other powders, both food and non-food, are pneumatically conveyed in industry. While specific results will depend on actual powder properties (size, shape, composition, etc.), the general effects of changing either operating parameters (conveying air speed) or pipeline configuration (pipe radius and bend radius) should have broad applicability.

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Figures.



Fig. 1 50 mm pipeline dilute phase pneumatic conveying rig.

1. Air source, 2. Electric vibrating feeder, 3. Funnel, 4. Vertical bend 1, 5. Vertical bend 26. Horizontal bend, 7. Sight glass, 8. Collector, 9. Pitot tube, 10. Pressure Meter.



Fig. 2 SEM micrograph of whey protein isolate (WPI, 90% Protein) powder

(a) WPI before conveying; (b) WPI after conveying at a superficial air velocity of 10 m/s; (c) WPI after conveying at 30 m/s where the scale bar has a length of 100 μ m, (d) WPI after conveying at 30 m/s with scale bar length of 20 μ m.



Fig. 3 Frequency particle size distribution (a) and Mean volume diameter D[4,3] (b) of WPI under different conveying air velocity.



Fig. 4 Effect of conveying air velocity (V) on the Particle size distribution of WPI in the cumulative form where a fixed solid feeding rate of 5 g/s is used.



Fig. 5 Effect of solid feeding rate (SL) on the particle size distribution of WPI powders both in frequencysize distribution and cumulative form.



Fig. 6 Three configurations of pipe bends.



Fig. 7 Effect of bend radius on the frequency-size distribution of WPI where conveying air velocity V = 20 m/s and solid feeding rate S = 5 g/s.



Fig. 8 Particle size distribution (PSD) of WPI before and after conveying with different initial particle size where conveying air velocity V = 20m/s and solid feeding rate S = 5g/s.



Fig. 9 Mean volume diameter D[4,3] of WPI powder with four initial PSD at different conveying airspeed with the same solid feeding rate S = 5g/s.



Fig. 10 Tapped bulk density of WPI powders under different conveying conditions.



Fig. 11 Powder flow function of WPI powders under different conveying conditions.



Fig. 12 Effect of operating conditions on the wettability of WPI powders.